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09/0140

PCT/EP 00/01738

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EP00/1738

REC'D	06 APR 2000
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Bescheinigung

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Attestation

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Patentanmeldung Nr. Patent application No. Demande de brevet n°

99200638.7

## PRIORITY DOCUMENT

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**Blatt 2 der Bescheinigung**  
**Sheet 2 of the certificate**  
**Page 2 de l'attestation**

Anmeldung Nr.:  
Application no.: 99200638.7  
Demande n°:

Anmeldetag:  
Date of filing: 05/03/99  
Date de dépôt:

Anmelder:  
Applicant(s):  
Demandeur(s):  
Akzo Nobel N.V.  
6824 BM Arnhem  
NETHERLANDS

Bezeichnung der Erfindung:  
Title of the invention:  
Titre de l'invention:

Esterquats, intermediates, process to make them, and their use

In Anspruch genommene Priorität(en) / Priority(ies) claimed / Priorité(s) revendiquée(s)

Staat:  
State:  
Pays:

Tag:  
Date:  
Date:

Aktenzeichen:  
File no.  
Numéro de dépôt:

Internationale Patentklassifikation:  
International Patent classification:  
Classification internationale des brevets:

C07C219/06, C07C219/08, C07C215/24, C11D1/62

Am Anmeldetag benannte Vertragsstaaten:  
Contracting states designated at date of filing: AT/BE/CH/CY/DE/DK/ES/FI/FR/GB/GR/IE/IT/LI/LU/MC/NL/PT/SE  
Etats contractants désignés lors du dépôt:

Bemerkungen:  
Remarks:  
Remarques:

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ESTERQUATS, INTERMEDIATES, PROCESS TO MAKE THEM, AND THEIR USE

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The Invention relates to specific quaternary ammonium compounds having at least one nitrogen-bonded moiety with at least one ester function, intermediates for making the compounds, compositions comprising one or more of these compounds, a process to make the compounds, as well as the use of such compounds as a fabric softener.

10

Quaternary ammonium compounds having a substituent on the nitrogen atom with two ester groups are known. Also compositions comprising such ammonium compounds are known. WO 97/47588, for instance, discloses how 2,3-dihydroxypropyl trimethyl ammonium chloride is reacted with lauric acid to form a composition comprising the corresponding diester quaternary compound, a propyl-diester-quat (PDQ).

15

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The use of PDQ as a fabric softener is known to result in superior softening performance. However, the manufacture of these compounds is cumbersome and the raw material N,N-dimethyl-1-amino propane-2,3-diol is costly. Also, the epichlorohydrin that is typically used to make this raw material for PDQ is less desired from an environmental viewpoint. Furthermore, it is noted that the production of PDQ typically requires the use of a solvent, such as Isopropanol, In the quaternization reaction. However, such solvent can transesterify with the

25

PDQ, resulting in the formation of a contaminant (the fatty acid ester of the solvent) and a reduction of the softening performance of the PDQ-containing product (because less diester compound is present). Therefore, there is a need for alternative compounds with a better price/performance that can be produced according to a process that puts less strain on the environment. Preferably, this

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process does not require the use of solvents that can transesterify with fatty acid esters.

Our investigations led us to a new and surprisingly simple process for making new type of quaternary compounds whereof at least one of the nitrogen substituents contains at least one ester group, and to new intermediates. These compounds, and compositions comprising these compounds, have a good fabric softening performance and a better price/performance than the conventional fabric softeners. It is noted that the intermediates may be used as a fabric softener by themselves. Also, the new process offers advantages from an environmental viewpoint because epichlorohydrin is not used. Furthermore, in one of the preferred embodiments, the quaternization step of the amine is not performed towards the end of the synthesis of the surface active compound, but already on the low molecular weight amine. Hereby, the process is improved, because it is much simpler to separate and purify the low molecular weight quaternized amine than to perform such steps on the resulting surface active compound, if so desired. More specifically, during processing/washing of a surface active compound, there is the tendency that the compound is being dispersed. In another preferred embodiment, a trialkylamine is reacted with an epoxy alkene in the presence of an acid, in order to produce a quaternary intermediate. The process of this preferred embodiment obviates the use of epichlorohydrin as well as the use of undesired solvents. The quaternary intermediate can be esterified to give the preferred diesterquat fabric softeners. However, also the monoesterquat that is formed can be useful as a fabric softener.

The new quaternary compounds according to the invention are of the formula:

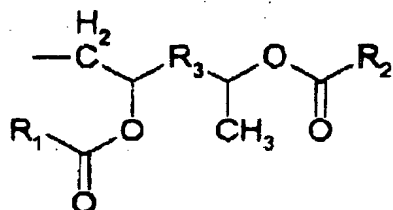




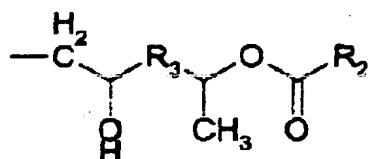
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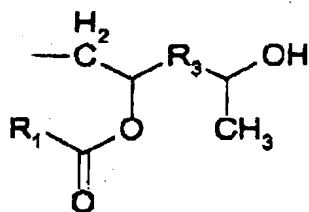
whereby Z is covalently bonded to the nitrogen atom and of the following formulae (I-IV)



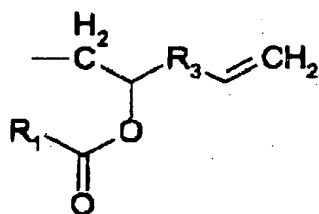
(I),



(II)

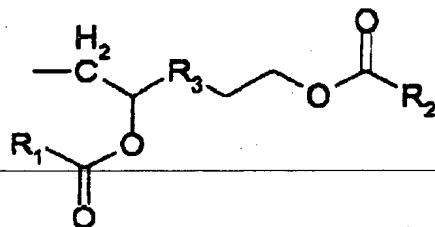


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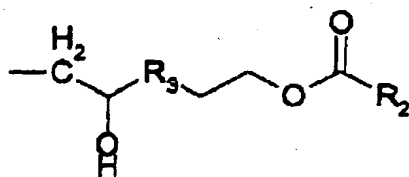


(IV)

or isomers thereof with formulae:



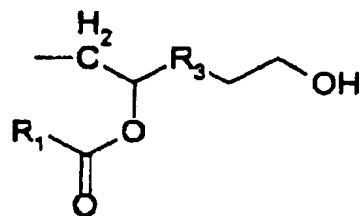
(Ia)



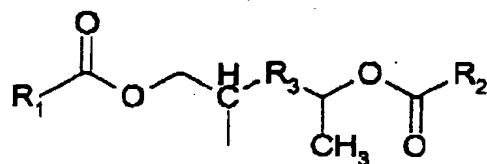
(IIa)

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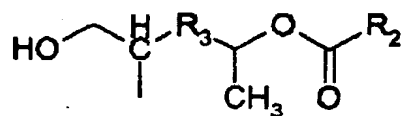
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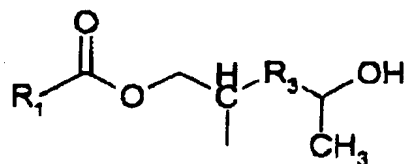
(IIIa)



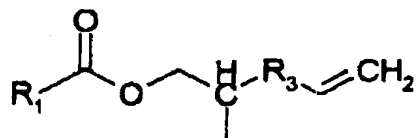
(Ib),



(IIb)

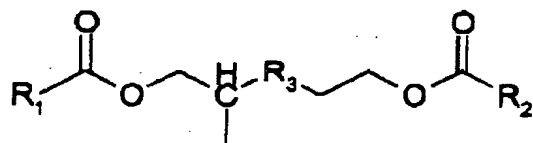


(IIIb)

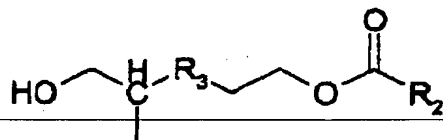


(IVb)

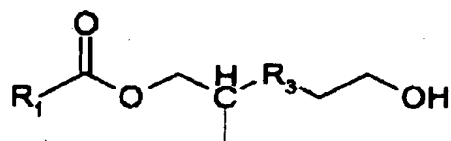
5



(Ic)



(IIc), and



(IIIc)

whereby,

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$R_1$  and  $R_2$  are independently selected from linear or branched, saturated or unsaturated  $C_{6-22}$  hydrocarbyl,

$R_3$  is nothing or  $C_{1-20}$  hydrocarbyl,

$R_4$  is  $C_{1-6}$  alkyl,  $C_{1-6}$  alkylene, or independent Z,

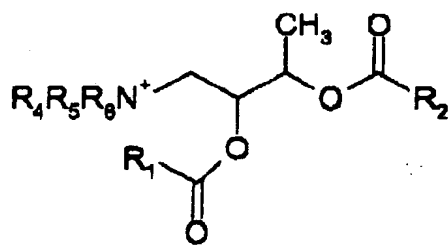
5  $R_5$  is H,  $C_{1-6}$  alkyl, independent Z, or the residue of the quaternizing agent, such as  $C_{1-30}$  alkyl or alkenyl, preferably,  $C_{1-7}$  alkyl or alkenyl,

$R_6$  is  $C_{1-6}$  alkyl or independent Z,

n is 1 or 2, and

$X^-$  is an ion selected from  $Cl^-$ ,  $Br^-$ ,  $I^-$ ,  $F^-$ ,  $CH_3SO_4^-$ ,  $C_2H_5SO_4^-$ ,  $H_2PO_4^-$ ,  $HPO_4^{2-}$ ,  $PO_4^{3-}$ ,  
10  $H_2PO_3^-$ ,  $HPO_3^{2-}$ ,  $H_2PO_2^-$ ,  $HPO_2^{2-}$ , nitrate<sup>-</sup>, formate<sup>-</sup>, acetate<sup>-</sup>, propionate<sup>-</sup>, tartrate<sup>-</sup> and benzoate<sup>-</sup>, whereby the total charge of the anions equals the total charge of the cations. The compounds may be used after purification and/or isolation. Preferably, they are part of a composition comprising more than one of the compounds of formula I-IV, and isomers thereof, since extensive isolation steps  
15 of the individual compounds can then be refrained from. Purification may include a bleaching and/or adsorption step to convert and/or remove chemicals that cause discoloration.

Preferred compounds are of the formula (I), written in full below



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$X^-$  (V)

or isomers thereof, whereby  $R_1$ - $R_6$  have the meaning as presented above.

More preferably, the compounds of the above formulae I-IV, or the isomers thereof, are of the formula wherein  $R_1$  and  $R_2$  are  $C_{12-18}$ . Even more preferably,

25  $R_1$  and  $R_2$  have a carbon distribution within said range as can be found in

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commercial fatty acids. Also, preferred compounds of formulae I-IV, or isomers thereof, are of the formula wherein  $R_4$  and  $R_5$  are methyl or ethyl.

Other preferred compounds of formulae I-IV, or isomers thereof, are of the formula wherein  $R_5$  is methyl or ethyl.

5 Preferably  $n$  is 1.

Further preferred compounds of formulae I-IV, or isomers thereof, are of the formula wherein  $X$  is chloride, methyl sulfate, or ethyl sulfate.

10 Another embodiment of the invention is the process to make the compounds according to formulae I-IV and/or isomers thereof. This process involves the reaction of an unsaturated epoxide with an amine, preferably a dialkylamine or trialkylamine, after which the unsaturated, hydroxy group substituted intermediate is reacted with, on average, 1-2 moles of fatty acid groups per mole of hydroxy group of the intermediate to form an ester. Using this ratio of  
15 fatty acid groups will ensure that at least part of the unsaturated bonds are reacted as well to form the preferred diesters.

It is noted that F.F. Blicke and J.H. Biel disclosed in J. Am.Chem.Soc. 79, 5508-5512, 1957, that 1,2-epoxy-3-butene can be reacted with aqueous  
20 dimethylamine hydrochloride to form 1-dimethylamino-2-hydroxy-3-butene. However, it is not disclosed that such a compound can be further reacted with 1-2 moles of fatty acid per mole of this product to form the compounds according to the invention with good fabric softening performance.

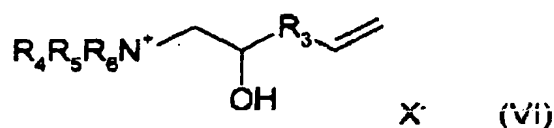
25 Another embodiment of the invention concerns amines that can be formed as intermediates by reacting a dialkylamine with an unsaturated epoxide, followed by esterification. These intermediates are of the formula  $R_4(R_5NZ)_n$ , wherein  $R_4$ ,  $R_5$ ,  $n$ , and  $Z$  have the meaning as specified above. These intermediates may be used as fabric softeners themselves. However, preferably they are quaternized

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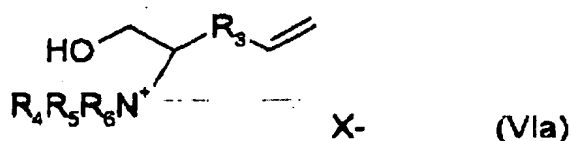
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In conventional ways with agents of formula  $R_3X$ , in order to give the preferred products of formula I-IV and isomers thereof.

- 5 A further embodiment of the invention is formed by the intermediate products that are formed by reacting a trialkylamine with an unsaturated epoxide in the presence of an acid. Preferably these compounds are of the formula



or the isomers thereof according to the formula



- 10 wherein  $R_3$ - $R_6$  and  $X$  have the meaning as specified above.

These intermediates can be easily transformed in the desired compounds of formula I-IV by direct or transesterification. The products of formula VI, or the isomers thereof, may themselves be used as a fabric softener. Therefore, a composition comprising compounds I-IV may also contain intermediates of

15 formula VI, without that the fabric softening properties are adversely affected.

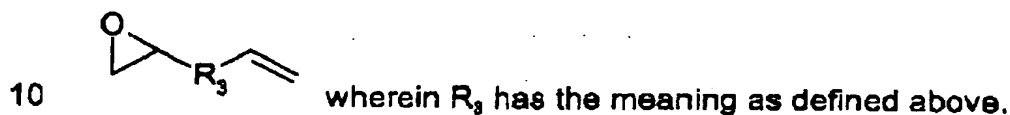
- A further embodiment of the present invention is the use of the compounds according to formulae I-VI, or the isomers thereof, as a fabric softener. Preferably, the compounds are of formula I or the isomers thereof. The
- 20 products are well-suited for this use because of their expected biodegradability, due to the two ester functions in the molecule, and their price/performance. It is noted that it is known in the art to further improve the performance of fabric softening compositions by combining the fabric softening compounds with a performance booster selected from the group consisting of cationic and non-
- 25 ionic surfactants. When used in this fashion, the fabric softening compounds are even more effectively deposited on the textile fabric.

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The amine that is reacted with the unsaturated epoxide is of the structure  $R_4[R_5R_6N]_n$ , or the protonated form thereof with structure  $R_4[R_5R_6N^+H]_n X^-$ , wherein  $R_4$ ,  $R_5$ ,  $R_6$ ,  $n$ , and  $X^-$  have the meaning as defined above. Preferably, a  
 5     dialkylamine or trialkylamine is reacted with the unsaturated epoxide. Preferably  $n$  is 1. Of amines with  $n$  is 2, ethylene diamine is preferred.

The unsaturated epoxide that can be used are preferably 1,2-epoxyalkenes of the formula



Preferred 1,2-epoxyalkenes are 1,2-epoxy-3-butene, 1,2-epoxy-4-pentene, 1,2-epoxy-5-hexene, 1,2-epoxy-6-heptene, 1,2-epoxy-3-pentene, 1,2-epoxy-3-hexene, 1,2-epoxy-3-heptene, and 1,2-epoxy-4-hexene. More preferred 1,2-epoxyalkenes are 1,2-epoxy-3-butene, 1,2-epoxy-3-pentene, 1,2-epoxy-3-hexene, and 1,2-epoxy-3-heptene. Most preferred is 1,2-epoxy-3-butene.  
 15

At least  $n$  mole of 1,2-epoxyalkene is to react with one mole of  $R_4[R_5R_6N]_n$ , or the protonated form thereof with structure  $R_4[R_5R_6N^+H]_n X^-$ , according to the invention. However, if a monoalkylamine or ammonia is used to make the  
 20     indicated dialkylamine or trialkylamine *in situ* by first reacting it with 1,2-epoxyalkene then in total up to four moles of 1,2-epoxyalkene can react per mole of amine.

25     The ester functions in the compounds of the invention typically are derived from saturated or unsaturated, linear or branched  $C_{6-30}$ , preferably  $C_{8-22}$ , more preferably  $C_{12-18}$ , most preferably naturally occurring, fatty acids, optionally substituted with, e.g., one or more hydroxy groups. Preferred fatty acids used in making the compounds of the invention include coconut, palm, palm kernel,

soya, oleic, tallow, rapeseed, canola, behenic, eruca fatty acids, and mixtures thereof. Preferably a fatty acid mixture is used comprising at least 50 percent by weight (%w/w) of  $C_{12-18}$  fatty acids.

The acids may be used as such in a conventional direct esterification process, but also derivatives can be used, such as the corresponding acid chlorides or (mixed) anhydrides. In transesterification reactions typically a fatty acid ester is used. In such transesterification reactions the methyl, ethyl, and/or glycerol esters of the acids are preferably used. Most preferred are mono-, di- and/or triglycerides of the acids.

Preferably 50-100 percent, on a molar basis, of the hydroxy functions of the 1-amino-2-hydroxy-alkene intermediate, that is formed after reacting amine and 1,2-epoxyalkene, are esterified. More preferably, 75-100 molar percent of the hydroxy groups is esterified. Also, preferably 50-100 percent, on a molar basis, of the unsaturated functions of the 1-amino-2-hydroxy-alkene intermediate is reacted with fatty acid to give an ester function. More preferably, 75-100 molar percent of the alkene functions is esterified.

If a dialkylamine was used in the reaction with the 1,2-epoxyalkene, then the resulting diester bearing amine is to be quaternized in order to achieve the preferred fabric softening compounds according to the invention. The quaternization step is conventional, using agents of formula  $R_5X$ , wherein  $R_5$  and  $X$  have the meaning as defined above. Examples of conventional quaternizing agents include, but are not limited to, dimethyl sulphate, diethyl sulphate, methyl chloride, methyl bromide, methyl iodide, benzyl chloride, benzyl bromide, allyl chloride, and allyl bromide.

However, in a preferred embodiment of the invention, the raw material is not a dialkylamine but a trialkylamine. When such trialkylamines are reacted with the unsaturated epoxide in the presence of a conventional activator for the ring

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opening of epoxides, typically an acid, such as hydrochloric acid, sulfuric acid, hydrochloric salts of amines, the hydrochloric salt of pyridine, and the like, then the corresponding quaternary ammonium group-bearing unsaturated alcohol is formed. These alcohols can be converted to the corresponding mono- and/or diesterquaternary ammonium compounds by appropriate esterification. The esterification can be a direct esterification or a transesterification, both processes being known in the art for conventional esterification processes. Preferably, C<sub>6-22</sub> fatty acid groups are introduced in said esterification process since such groups are needed to obtain the desired fabric softening effect.

10

The resulting quaternary ammonium compounds, having a nitrogen substituent with at least one ester group, can be used as is or after purification and/or isolation. The compounds, preferably the diester-bearing compounds, are preeminently suited for use as a biodegradable fabric softener.

15

The invention is elucidated by the following, non-optimized, examples.

#### Materials used:

Hypophosphorous acid ex Aldrich

20 Stearic acid ex Merck

Dimethylaminobutenol ex Eastman

Butadiene monoxide (1,2-epoxy-3-butene) ex Aldrich

#### Procedure

25 A mixture of N-(2-hydroxybut-3-en-1-yl)-N,N,N-trimethylammonium chloride and of N-(1-hydroxybut-3-en-2-yl)-N,N,N-trimethylammonium chloride was produced by charging a 10 ml. flask, equipped with a stirrer, with a mixture of 2.55g (26.7 mmol) trimethylaminehydrochloride, 0.06g (0.5 mmol) of 47.7 %w/w of trimethylamine in water and 2g water and cooling the mixture to 10°C. Then



2.09g (29.9 mmol) of 1,2-epoxybutene was added dropwise during 1 hour while the temperature was maintained at 10°C. Thereafter, the mixture was heated to 40°C and further reacted for 2 hours. Subsequently, 1 ml of 1M HCl was added after which the water was evaporated at atmospheric pressure. The desired  
5 mixture remained.

### Example 1

in a one necked flask of 100ml, equipped with stirrer and water separator, 1.58 g (10.2 mmole) of a mixture of N,N-dimethyl-1-aminobut-3-en-2-ol hydrochloride  
10 and N,N-dimethyl-2-aminobut-3-en-1-ol hydrochloride with a purity of 97.5 % by weight (%w/w) was combined with 6.07 g (21.4 mmole) stearic acid. To the mixture, 1 ml of 50 %w/w of hypophosphorous acid in water was added as a catalyst. The mixture was stirred for 4 hours at 160°C at a reduced pressure of 1 mbar.

15 A mixture of 2,3-di(stearoyloxy)but-1-yl)dimethylaminehydrochloride and 1,3-di(stearoyloxy)but-2-yl)dimethylaminehydrochloride was formed with a yield of 24 mole%, based on the amino compound.

The product can be quaternized in conventional ways.

### 20 Example 2

In a one necked flask of 100ml, equipped with stirrer and water separator, 1 ml of 50% by weight hypophosphorous acid in water was added to a mixture of 1.71g (10.3 mmol) N-(2-hydroxybut-3-en-1-yl)-N,N,N-trimethylammonium  
chloride / N-(1-hydroxybut-3-en-2-yl)-N,N,N-trimethylammonium chloride and  
25 5.96g (21.0 mmol) stearic acid. While stirring, the mixture was reacted at 160°C for 4 hours under vacuum at a pressure of 1.33 mbar.

A mixture of 2,3-di(stearoyloxy)but-1-yl)trimethyl ammonium chloride (or 1-N,N,N-trimethylammoniumchloride-butane-2,3-distearate) and 1-3-di-

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(stearyloxy)but-2-yl)trimethylammonium chloride was formed with a yield of 49 mole%, based on the ammonium compound.

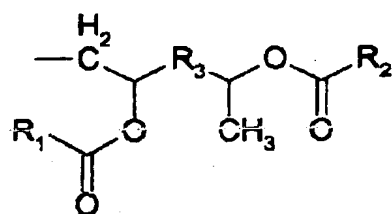
The product has good fabric softening properties.

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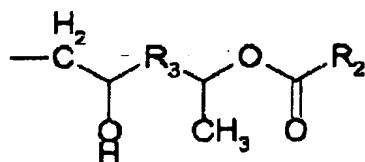
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# Claims

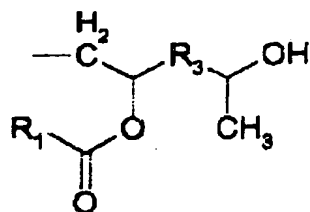
1. Quaternary ammonium compounds of the formula  $R_4[R_3R_5N^+Z]_n X^-$ , whereby Z is covalently bonded to the nitrogen atom and selected from the group of following formulae (I-IV)



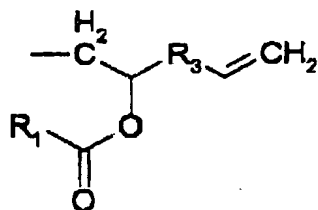
(I),



(II),



(III),



(IV),

- 10 and isomers thereof, whereby  
 $R_1$  and  $R_2$  are independently selected from linear or branched, saturated or unsaturated  $C_{6-22}$  hydrocarbyl,  
 $R_3$  is nothing or  $C_{1-20}$  hydrocarbyl,  
 $R_4$  is  $C_{1-8}$  alkyl,  $C_{1-8}$  alkylene, or independent Z,

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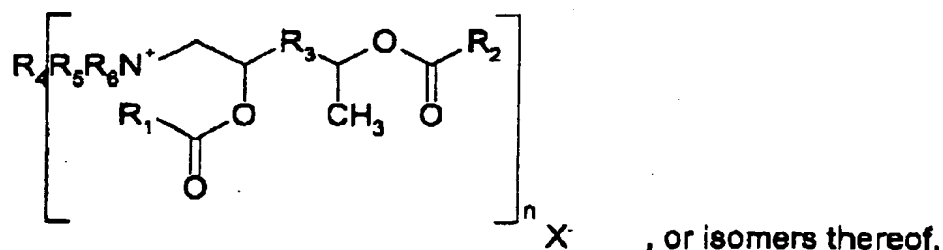
$R_5$  is H,  $C_{1-6}$  alkyl, independent Z, or the residue of the quaternizing agent, such as  $C_{1-30}$  alkyl or alkenyl, preferably,  $C_{1-7}$  alkyl or alkenyl,

$R_6$  is  $C_{1-6}$  alkyl or independent Z,

n is 1 or 2, and

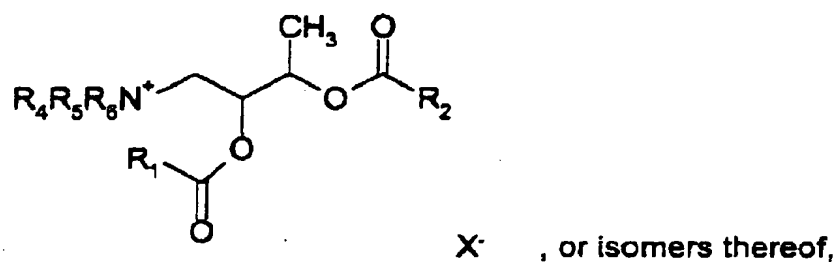
- 5  $X^-$  is an ion selected from  $Cl^-$ ,  $Br^-$ ,  $I^-$ ,  $F^-$ ,  $CH_3SO_4^-$ ,  $C_2H_5SO_4^-$ ,  $H_2PO_4^-$ ,  $HPO_4^{2-}$ ,  $PO_4^{3-}$ ,  $H_2PO_3^-$ ,  $HPO_3^{2-}$ ,  $H_2PO_2^-$ ,  $HPO_2^{2-}$ , nitrate<sup>-</sup>, formate<sup>-</sup>, acetate<sup>-</sup>, propionate<sup>-</sup>, tartrate<sup>-</sup> and benzoate<sup>-</sup>, whereby the total charge of the anions equals the total charge of the cations.

- 10 2. Compounds according to claim 1 of the formula



wherein  $R_1$ - $R_6$ , n, and  $X^-$  have the meaning as given in claim 1.

3. Compounds according to claim 2 of formula



wherein  $R_1$ ,  $R_2$ ,  $R_4$ - $R_6$  and  $X^-$  have the meaning as given in claim 1.

4. Compounds according to any one of claims 1-3 wherein  $R_1$  and  $R_2$  are independently selected from linear or branched, saturated or unsaturated  $C_{12-18}$  alkyl groups.

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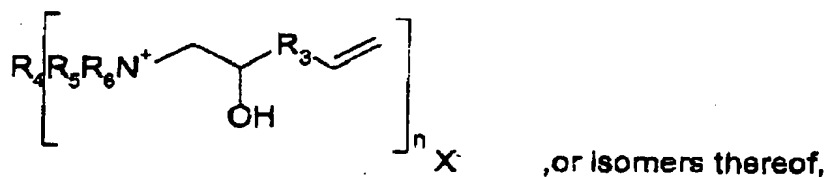
5. Compounds according to any one of the preceding claims, characterized in that  $R_4$  and  $R_6$  are methyl.

5 6. Compounds according to any one of the preceding claims wherein  $X^-$  is chloride, methyl sulfate or ethyl sulfate.

7. Intermediates for making one or more of the compounds of the previous claims according to the formula  $R_4[R_6NZ]_n$ , wherein  $R_4$ ,  $R_6$ ,  $n$ , and  $Z$  have the meaning given in claim 1.

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8. Intermediates for making one or more of the compounds of claims 1-6 according to the formula



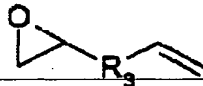
wherein  $R_3$ - $R_6$ ,  $n$ , and  $X$  have the meaning given in claim 1.

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9. Compositions comprising one or more of the compounds according to any of the previous claims.

10. Process to make compounds of claim 1 comprising the steps of:

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- reacting an unsaturated epoxide of formula  with an amine or protonated amine, of formulae  $R_4[R_5R_6N]_n$  or  $R_4[R_5R_6N^+H]_n X^-$ , wherein  $R_3$ ,  $R_4$ ,  $R_5$ ,  $R_6$ ,  $n$ , and  $X^-$  have the meaning as given in claim 1, and

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- esterification of the intermediate with, on average, 1-2 moles of fatty acid derivatives, comprising moieties  $R_1-C(O)-$ ,  $R_2-C(O)-$  or mixtures thereof, per mole of OH groups of the intermediate,
- an optional conventional quaternization either before or after said esterification step.

5

11. Process according to claim 10, characterized in that a trialkylamine is reacted with the unsaturated epoxide.

10 12. Process according to claims 10 or 11, characterized in that a product according to any one of claims 2-6 is formed.

13. Use of a compound or composition according to any one of claims 1-9 as a fabric softener.

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14. Use according to claim 14 wherein the compound is used in combination with a conventional performance booster selected from the group consisting of cationic and non-ionic surfactants.

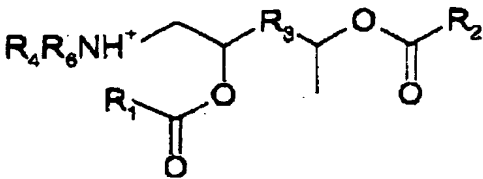
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Abstract

Products, intermediates, a process to make such products, and the use of such products as a fabric softener is presented whereby said products are preferably



5 of formula  $X^-$  or isomers thereof,

whereby  $R_1$  and  $R_2$  are independently selected from linear or branched, saturated or unsaturated  $C_{6-22}$  hydrocarbyl,

$R_3$  is nothing or  $C_{1-20}$  hydrocarbyl,

$R_4$  is  $C_{1-8}$  alkyl or independent Z,

10  $R_5$  is H,  $C_{1-8}$  alkyl, independent Z, or the residue of the quaternizing agent, such as  $C_{1-30}$  alkyl or alkenyl, preferably,  $C_{1-7}$  alkyl or alkenyl,

$R_6$  is  $C_{1-8}$  alkyl or independent Z, and

$X^-$  is an ion selected from  $Cl^-$ ,  $Br^-$ ,  $I^-$ ,  $F^-$ ,  $CH_3SO_3^-$ ,  $C_2H_5SO_3^-$ ,  $H_2PO_4^-$ ,  $HPO_4^{2-}$ ,  $PO_4^{3-}$ ,  $H_2PO_3^-$ ,  $HPO_3^{2-}$ ,  $H_2PO_2^-$ ,  $HPO_2^{2-}$ , nitrate, formate, acetate, propionate, tartrate

15 and benzoate, whereby the total charge of the anions equals the total charge of the cations and said process concerns the reaction of an unsaturated epoxide with an amine or a protonated amine, esterification of the formed intermediate, and optional quaternization.

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